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# SILVERMAN 13273-13283

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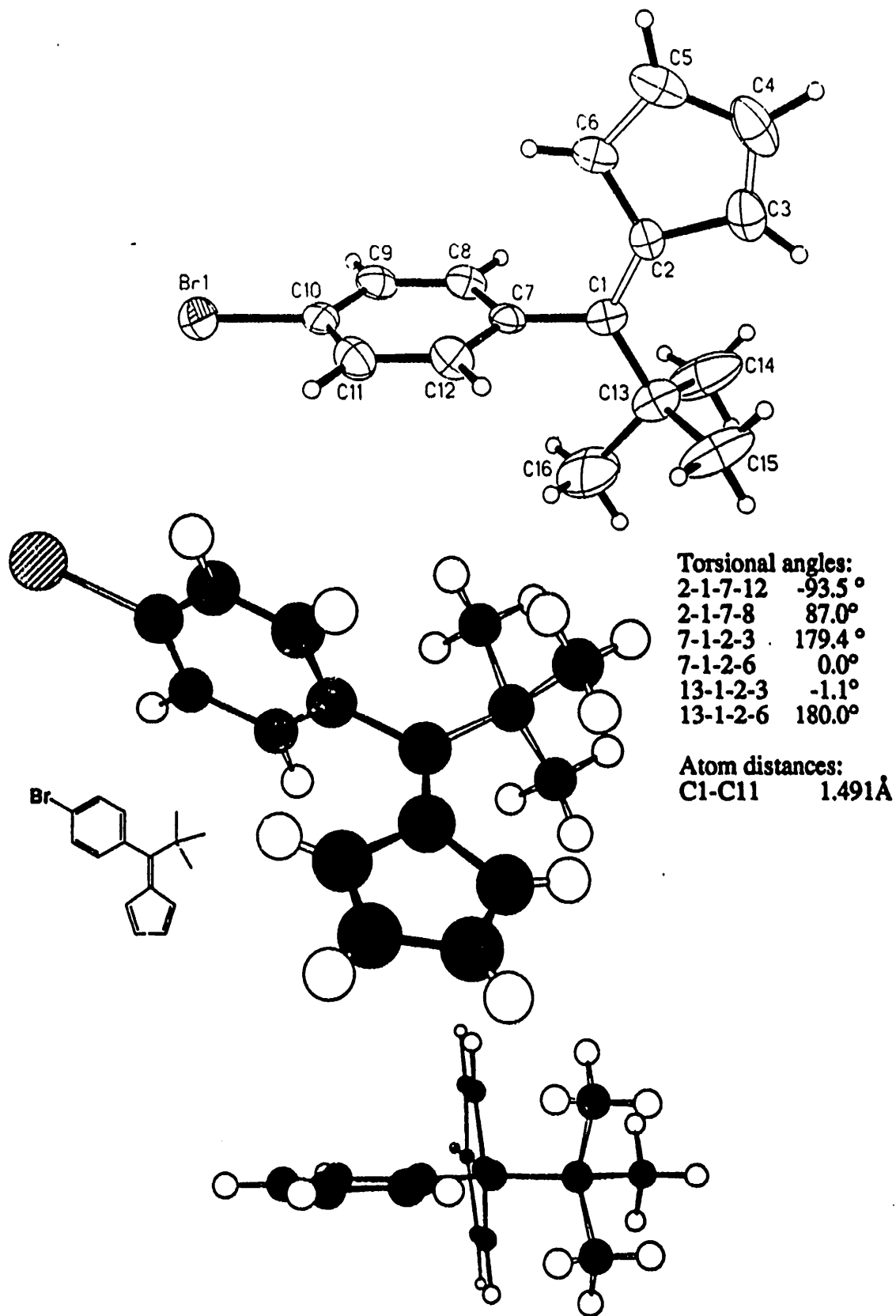


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H-13283-m1

6-(*p*-Bromophenyl)-6-*t*-butylfulvene (9)



Collection of X-ray Diffraction Data. A yellow crystal of approximate dimensions 0.10 x 0.20 x 0.36 mm was oil-mounted on a glass fiber and transferred to the Syntex P2<sub>1</sub> automated four-circle diffractometer which is equipped with a modified LT-1 low temperature system. The determination of Laue symmetry, crystal class, unit cell parameters and the crystal's orientation matrix were carried out by previously described methods similar to those of Churchill<sup>1</sup>. Intensity data were collected at 168 K using a  $\theta$ -2 $\theta$  scan technique with Mo K $\alpha$  radiation under the conditions described in Table 1. All 2079 data were corrected for absorption and for Lorentz and polarization effects and were placed on an approximately absolute scale. The diffraction symmetry was 2/m with systematic absences 0k0 for  $k = 2n+1$  and  $h0l$  for  $l = 2n+1$ . The centrosymmetric monoclinic space group P2<sub>1</sub>/c [C<sub>2h</sub><sup>5</sup>; No. 14] is therefore uniquely defined.

Solution and Refinement of the Crystal Structure. All crystallographic calculations were carried out using either our locally modified version of the UCLA Crystallographic Computing Package<sup>2</sup> or the SHELXTL PLUS program set<sup>3</sup>. The analytical scattering factors for neutral atoms were used throughout the analysis<sup>4a</sup>; both the real ( $\Delta f'$ ) and imaginary ( $i\Delta f''$ ) components of anomalous dispersion<sup>4b</sup> were included. The quantity minimized during least-squares analysis was  $\sum w(|F_o| - |F_c|)^2$  where  $w^{-1} = \sigma^2(|F_o|) + 0.0005(|F_o|)^2$ .

The structure was solved by direct methods (SHELXTL PLUS) and refined by full-matrix least-squares techniques. Hydrogen atoms were included using a riding model with  $d(C-H) = 0.96\text{\AA}$  and  $U(\text{iso}) = 0.08\text{\AA}^2$ . Refinement of positional and thermal parameters led to convergence with  $R_F = 4.8\%$ ;  $R_{wF} = 5.0\%$  and GOF = 1.40 for 155 variables refined against those 1437 data with  $|F_o| > 3.0\sigma(|F_o|)$ . A final difference-Fourier synthesis showed no significant features,  $\rho(\text{max}) = 0.52\text{e}\text{\AA}^{-3}$ .

Table 1. Experimental Data for the X-ray Diffraction Study

H-13283-m3

Formula:  $C_{16}H_{17}Br$

Fw: 289.2

Temperature (K): 168

Crystal System: Monoclinic

Space Group:  $P2_1/c$  [ $C_{2h}^5$ ; No. 14]

$a = 9.3008(7) \text{ \AA}$

$b = 16.2059(11) \text{ \AA}$

$c = 9.6876(8) \text{ \AA}$

$\beta = 105.155(6)^\circ$

$V = 1409.4(2) \text{ \AA}^3$

$Z = 4$

$D_{\text{calcd}}, \text{ Mg/m}^3 = 1.363$

Diffractometer: Syntex  $P2_1$  (Siemens R3m/V System).

Radiation: Mo  $K\alpha$  ( $\lambda = 0.710730 \text{ \AA}$ )

Monochromator: Highly oriented graphite

Data Collected:  $+h, +k, \pm l$

Scan Type:  $\theta$ - $2\theta$

Scan Width:  $1.2^\circ$  plus  $K\alpha$ -separation

Scan Speed:  $3.0 \text{ deg min}^{-1}$  (in  $\omega$ )

$2\theta$  Range, deg: 4.0 to 45.0

$\mu(\text{Mo } K\alpha), \text{ mm}^{-1} = 2.86$

Absorption Correction: Semi-empirical ( $\psi$ -scan method)

Reflections Collected: 2079

Reflections with  $|F_o| > 3.0\sigma(|F_o|)$ : 1437

No. of Variables: 155

$R_F = 4.8\%$ ,  $R_{wF} = 5.0\%$

Goodness of Fit: 1.14

Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement coefficients ( $\text{\AA}^2 \times 10^4$ )

14-13283-my

|       | x        | y        | z       | U(eq)   |
|-------|----------|----------|---------|---------|
| C(1)  | 9536(5)  | 10334(3) | 2358(5) | 258(19) |
| C(2)  | 11007(6) | 10128(3) | 2738(5) | 270(19) |
| C(3)  | 12344(7) | 10643(4) | 3020(6) | 433(24) |
| C(4)  | 13520(7) | 10159(5) | 3338(6) | 568(28) |
| C(5)  | 13052(6) | 9300(4)  | 3285(6) | 493(25) |
| C(6)  | 11562(6) | 9272(3)  | 2929(5) | 340(20) |
| C(7)  | 8435(6)  | 9646(3)  | 2131(5) | 271(18) |
| C(8)  | 7971(6)  | 9309(3)  | 3275(5) | 342(20) |
| C(9)  | 6971(5)  | 8665(3)  | 3082(5) | 337(20) |
| C(10) | 6414(5)  | 8348(3)  | 1715(6) | 309(20) |
| C(11) | 6842(6)  | 8673(3)  | 553(6)  | 376(21) |
| C(12) | 7848(6)  | 9316(3)  | 773(5)  | 368(21) |
| C(13) | 8916(7)  | 11218(3) | 2152(6) | 402(22) |
| C(14) | 9291(10) | 11686(4) | 3523(7) | 905(38) |
| C(15) | 9352(9)  | 11662(4) | 966(7)  | 776(34) |
| C(16) | 7161(8)  | 11200(4) | 1665(9) | 890(39) |
| Br(1) | 5044(1)  | 7461(1)  | 1433(1) | 481(3)  |

\* Equivalent isotropic U defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor

Table 3. Interatomic Distances (Å) with Esd's

H-13283-m5

|             |           |             |           |
|-------------|-----------|-------------|-----------|
| C(1)-C(2)   | 1.362(7)  | C(1)-C(7)   | 1.491(7)  |
| C(1)-C(13)  | 1.536(7)  | C(2)-C(3)   | 1.463(8)  |
| C(2)-C(6)   | 1.474(7)  | C(3)-C(4)   | 1.316(9)  |
| C(4)-C(5)   | 1.456(10) | C(5)-C(6)   | 1.338(8)  |
| C(7)-C(8)   | 1.401(8)  | C(7)-C(12)  | 1.394(7)  |
| C(8)-C(9)   | 1.376(7)  | C(9)-C(10)  | 1.388(7)  |
| C(10)-C(11) | 1.391(8)  | C(10)-Br(1) | 1.893(5)  |
| C(11)-C(12) | 1.379(7)  | C(13)-C(14) | 1.490(8)  |
| C(13)-C(15) | 1.499(9)  | C(13)-C(16) | 1.577(10) |

Table 4. Interatomic Angles (Deg.) with Esd's

|                   |          |                   |          |
|-------------------|----------|-------------------|----------|
| C(2)-C(1)-C(7)    | 117.3(4) | C(2)-C(1)-C(13)   | 125.4(5) |
| C(7)-C(1)-C(13)   | 117.2(4) | C(1)-C(2)-C(3)    | 130.9(5) |
| C(1)-C(2)-C(6)    | 124.0(5) | C(3)-C(2)-C(6)    | 105.1(5) |
| C(2)-C(3)-C(4)    | 108.5(6) | C(3)-C(4)-C(5)    | 109.9(6) |
| C(4)-C(5)-C(6)    | 108.7(5) | C(2)-C(6)-C(5)    | 107.9(5) |
| C(1)-C(7)-C(8)    | 121.2(4) | C(1)-C(7)-C(12)   | 120.7(5) |
| C(8)-C(7)-C(12)   | 118.1(5) | C(7)-C(8)-C(9)    | 121.7(5) |
| C(8)-C(9)-C(10)   | 118.7(5) | C(9)-C(10)-C(11)  | 121.1(5) |
| C(9)-C(10)-Br(1)  | 119.2(4) | C(11)-C(10)-Br(1) | 119.6(4) |
| C(10)-C(11)-C(12) | 119.1(5) | C(7)-C(12)-C(11)  | 121.2(5) |
| C(1)-C(13)-C(14)  | 111.5(4) | C(1)-C(13)-C(15)  | 112.3(5) |
| C(14)-C(13)-C(15) | 113.0(5) | C(1)-C(13)-C(16)  | 110.2(5) |
| C(14)-C(13)-C(16) | 105.0(6) | C(15)-C(13)-C(16) | 104.2(5) |

H-13283-m6

Table 5. Anisotropic displacement coefficients ( $\text{\AA}^2 \times 10^4$ )

|       | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|-------|----------|----------|----------|----------|----------|----------|
| C(1)  | 340(34)  | 324(29)  | 150(27)  | 61(25)   | 136(25)  | 2(21)    |
| C(2)  | 305(35)  | 364(31)  | 160(28)  | -74(26)  | 92(23)   | -14(22)  |
| C(3)  | 452(42)  | 559(37)  | 265(34)  | -180(34) | 54(29)   | 5(27)    |
| C(4)  | 368(42)  | 1006(57) | 302(35)  | -239(41) | 40(29)   | 90(33)   |
| C(5)  | 310(38)  | 760(47)  | 392(37)  | 114(34)  | 63(28)   | 56(31)   |
| C(6)  | 290(34)  | 429(33)  | 298(31)  | 82(27)   | 71(25)   | 7(25)    |
| C(7)  | 190(28)  | 306(28)  | 308(32)  | 63(25)   | 52(24)   | 46(24)   |
| C(8)  | 311(33)  | 414(32)  | 288(32)  | 43(28)   | 53(25)   | 33(24)   |
| C(9)  | 240(31)  | 444(33)  | 340(33)  | 38(27)   | 102(25)  | 87(26)   |
| C(10) | 159(29)  | 298(30)  | 472(35)  | 57(24)   | 84(25)   | 51(25)   |
| C(11) | 326(33)  | 417(35)  | 362(34)  | 13(28)   | 51(26)   | -76(26)  |
| C(12) | 342(35)  | 457(33)  | 308(34)  | -3(29)   | 89(26)   | -22(26)  |
| C(13) | 561(41)  | 360(32)  | 318(33)  | 98(29)   | 174(29)  | -6(26)   |
| C(14) | 1655(82) | 483(44)  | 532(46)  | 489(48)  | 203(49)  | 27(33)   |
| C(15) | 1231(67) | 571(45)  | 634(48)  | 373(45)  | 433(45)  | 321(35)  |
| C(16) | 775(61)  | 548(45)  | 1322(76) | 341(44)  | 231(53)  | 139(44)  |
| Br(1) | 354(4)   | 405(4)   | 646(5)   | -42(3)   | 61(3)    | 46(3)    |

The anisotropic displacement exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + \dots + 2hka^*b^*U_{12})$$

Table 6. H-Atom coordinates ( $\times 10^4$ ) and isotropic displacement coefficients ( $\text{\AA}^2 \times 10^4$ )

H-13283-m7 7

|        | x     | y     | z    | U   |
|--------|-------|-------|------|-----|
| H(3A)  | 12373 | 11234 | 2972 | 800 |
| H(4A)  | 14537 | 10342 | 3581 | 800 |
| H(5A)  | 13706 | 8831  | 3463 | 800 |
| H(6A)  | 10956 | 8785  | 2818 | 800 |
| H(8A)  | 8389  | 9524  | 4218 | 800 |
| H(9A)  | 6636  | 8450  | 3867 | 800 |
| H(11A) | 6408  | 8458  | -387 | 800 |
| H(12A) | 8166  | 9534  | -21  | 800 |
| H(14A) | 10355 | 11715 | 3875 | 800 |
| H(14B) | 8889  | 12234 | 3368 | 800 |
| H(14C) | 8884  | 11409 | 4212 | 800 |
| H(15A) | 10419 | 11692 | 1182 | 800 |
| H(15B) | 8979  | 11364 | 89   | 800 |
| H(15C) | 8944  | 12210 | 866  | 800 |
| H(16A) | 6795  | 11756 | 1535 | 800 |
| H(16B) | 6829  | 10902 | 782  | 800 |
| H(16C) | 6790  | 10935 | 2389 | 800 |



STRUCTURE DETERMINATION SUMMARY

H-13283-m8

Crystal Data

|                        |   |
|------------------------|---|
| Empirical Formula      | $C_{16}H_{17}Br$  |
| Color; Habit           | Yellow prism  |
| Crystal Size (mm)      | 0.10 x 0.20 x 0.36  |
| Crystal System         | Monoclinic  |
| Space Group            | $P2_1/c$  |
| Unit Cell Dimensions   | $a = 9.3008(7) \text{ \AA}$<br>$b = 16.2059(11) \text{ \AA}$<br>$c = 9.6876(8) \text{ \AA}$<br>$\beta = 105.155(6)^\circ$ |
| Volume                 | $1409.4(2) \text{ \AA}^3$   |
| Z                      | 4   |
| Formula weight         | 289.2   |
| Density(calc.)         | $1.363 \text{ Mg/m}^3$  |
| Absorption Coefficient | $2.864 \text{ mm}^{-1}$   |
| F(000)                 | 592   |

Data Collection

H-13283-mg

|                         |   |
|-------------------------|---|
| Diffractometer System   | Siemens R3m/V   |
| Radiation               | MoK $\alpha$ ( $\lambda$ = 0.71073 Å)                           |
| Temperature (K)         | 168   |
| Monochromator           | Highly oriented graphite crystal                                |
| 2 $\theta$ Range        | 4.0 to 45.0°  |
| Scan Type               | $\theta$ -2 $\theta$  |
| Scan Speed              | Fixed; 3.00°/min. in $\omega$                                   |
| Scan Range ( $\omega$ ) | 1.20° plus K $\alpha$ -separation                               |
| Background Measurement  | Estimated from 96 step profile                                  |
| Standard Reflections    | 2 measured every 98 reflections                                 |
| Index Ranges            | $0 \leq h \leq 10$ , $0 \leq k \leq 17$<br>$-10 \leq l \leq 10$ |
| Reflections Collected   | 2079  |
| Independent Reflections | 1696 ( $R_{\text{int}}$ = 1.5%); ( $ F_o  > 0$ )                |
| Observed Reflections    | 1437 ( $ F_o  > 3.0\sigma( F_o )$ )                             |
| Absorption Correction   | Semi-empirical ( $\psi$ -scan method)                           |
| Min./Max. Transmission  | 0.3883 / 0.4917   |

Solution and Refinement

10  
H-13283-m10

|                                  |  |
|----------------------------------|--|
| System Used                      | Siemens SHELXTL (MicroVAX & PC Versions)   |
| Solution                         | Direct Methods   |
| Refinement Method                | Full-Matrix Least-Squares  |
| Quantity Minimized               | $\sum w( F_o  -  F_c )^2$  |
| Extinction Correction            | $\chi = 0.0004(2)$ , where<br>$F^* = F [ 1 + 0.002\chi F^2 / \sin(2\theta) ]^{-1/4}$ |
| Hydrogen Atoms                   | Riding model, fixed isotropic U  |
| Weighting Scheme                 | $w^{-1} = \sigma^2( F_o ) + 0.0005( F_o )^2$   |
| Final R Indices (obs. data)      | $R_F = 4.8\%$ , $R_{wF} = 5.0\%$   |
| Goodness-of-Fit                  | 1.40   |
| Number of Variables              | 155  |
| Data-to-Parameter Ratio          | 9.3:1  |
| Largest and Mean $\Delta/\sigma$ | 0.001, < 0.001   |
| Largest Difference Peak          | 0.52 eÅ <sup>-3</sup>  |
| Largest Difference Hole          | -0.30 eÅ <sup>-3</sup>   |

H-13283-m11 11

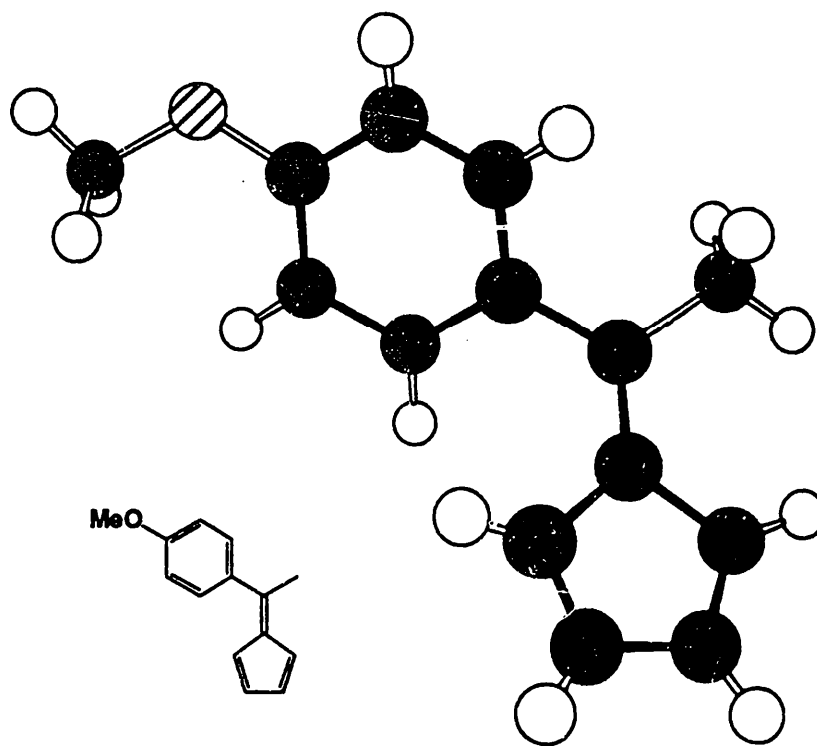
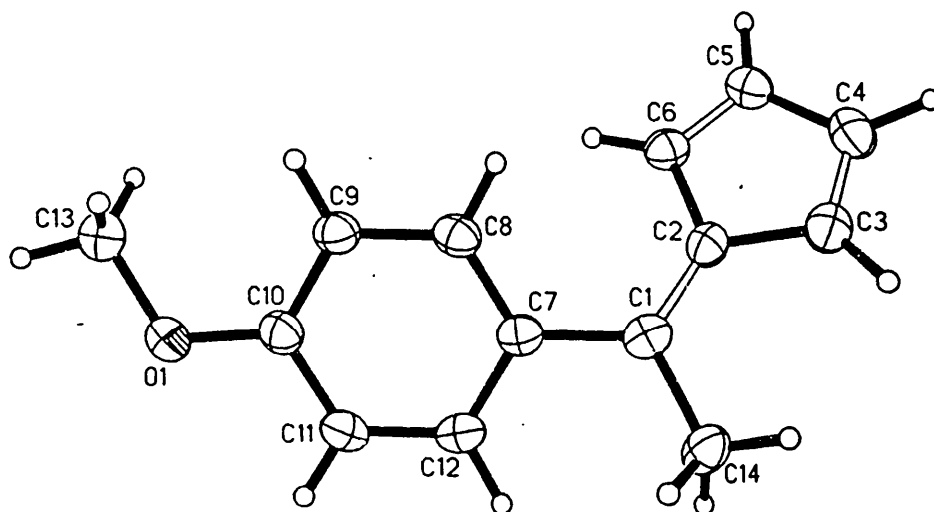
References.

1. Churchill, M. R.; Lashewycz, R. A.; Rotella, F. J. Inorg. Chem. 1977, 16, 265-271.
2. UCLA Crystallographic Computing Package, University of California Los Angeles, 1981, C. Strouse; personal communication.
3. Siemens Analytical X-Ray Instruments, Inc.; Madison, WI 1990.
4. International Tables for X-Ray Crystallography; Kynoch Press: Birmingham, England, 1974; (a) pp 99-101; (b) pp 149-150.

\* The thermal ellipsoid plot is shown at the 50% probability level.

H-13283-m12

6-(*p*-Methoxyphenyl)-6-methylfulvene (8)

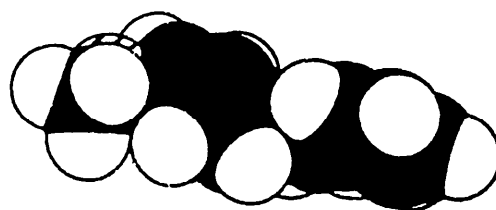
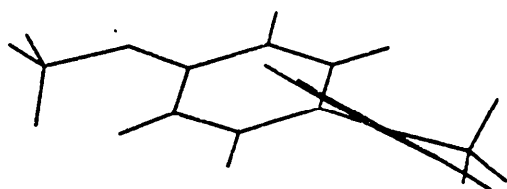


Torsional angles:

|          |        |
|----------|--------|
| 2-1-7-8  | -38.7° |
| 2-1-7-12 | 144.0° |
| 7-1-2-3  | 172.0° |
| 7-1-2-6  | -13.3° |
| 14-1-2-3 | -9.8°  |
| 14-1-2-6 | 165.0° |

Atom distances:

|       |         |
|-------|---------|
| C1-C7 | 1.479 Å |
| H6-H8 | 2.366 Å |



Collection of X-ray Diffraction Data. An orange crystal of approximate dimensions  $0.23 \times 0.30 \times 0.52$  mm was oil-mounted on a glass fiber and transferred to the Syntex P2<sub>1</sub> automated four-circle diffractometer which is equipped with a modified LT-1 low temperature system. The determination of Laue symmetry, crystal class, unit cell parameters and the crystal's orientation matrix were carried out by previously described methods similar to those of Churchill<sup>1</sup>. Intensity data were collected at 168 K using a  $\theta$ - $2\theta$  scan technique with Mo K $\alpha$  radiation under the conditions described in Table 1. All 2107 data were corrected for Lorentz and polarization effects and were placed on an approximately absolute scale. The diffraction symmetry was 2/m with systematic absences  $0k0$  for  $k = 2n+1$  and  $h0l$  for  $l = 2n+1$ . The centrosymmetric monoclinic space group P2<sub>1</sub>/c [C<sub>2h</sub><sup>5</sup>; No. 14] is therefore uniquely defined.

Solution and Refinement of the Crystal Structure. All crystallographic calculations were carried out using either our locally modified version of the UCLA Crystallographic Computing Package<sup>2</sup> or the SHELXTL PLUS program set<sup>3</sup>. The analytical scattering factors for neutral atoms were used throughout the analysis<sup>4a</sup>; both the real ( $\Delta f'$ ) and imaginary ( $i\Delta f''$ ) components of anomalous dispersion<sup>4b</sup> were included. The quantity minimized during least-squares analysis was  $\sum w(|F_o| - |F_c|)^2$  where  $w^{-1} = \sigma^2(|F_o|) + 0.0005(|F_o|)^2$ .

The structure was solved by direct methods (SHELXTL PLUS) and refined by full-matrix least-squares techniques. Hydrogen atoms were located from a difference-Fourier map and included with isotropic temperature parameters. Refinement of positional and thermal parameters led to convergence with  $R_F = 3.6\%$ ;  $R_{wF} = 4.5\%$  and GOF = 1.51 for 193 variables refined against those 1630 data with  $|F_o| > 3.0\sigma(|F_o|)$ . A final difference-Fourier synthesis showed no significant features,  $\rho(\max) = 0.19\text{e}\text{\AA}^{-3}$ .

Table 1. Experimental Data for the X-ray Diffraction Study

14  
H-13283-m14

Formula:  $C_{14}H_{14}O$

Fw: 198.3

Temperature (K): 168

Crystal System: Monoclinic

Space Group:  $P2_1/c$  [ $C_{2h}^5$ ; No. 14]

$a = 10.7334(9) \text{ \AA}$

$b = 11.1897(9) \text{ \AA}$

$c = 9.5808(9) \text{ \AA}$

$\beta = 112.265(7)^\circ$

$V = 1064.89(16) \text{ \AA}^3$

$Z = 4$

$D_{\text{calcd}}, \text{ Mg/m}^3 = 1.237$

Diffractometer: Syntex  $P2_1$  (Siemens R3m/V System)

Radiation: Mo  $K\alpha$  ( $\bar{\lambda} = 0.710730 \text{ \AA}$ )

Monochromator: Highly oriented graphite

Data Collected:  $+h, +k, \pm l$

Scan Type:  $\theta-2\theta$

Scan Width:  $1.2^\circ$  plus  $K\alpha$ -separation

Scan Speed:  $3.0 \text{ deg min}^{-1}$  (in  $\omega$ )

$2\theta$  Range, deg: 4.0 to 50.0

$\mu(\text{Mo } K\alpha), \text{ mm}^{-1} = 0.071$

Reflections Collected: 2107

Reflections with  $|F_o| > 3.0\sigma(|F_o|)$ : 1630

No. of Variables: 193

$R_F = 3.6\%$ ,  $R_{wF} = 4.5\%$

Goodness of Fit: 1.51

Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement coefficients ( $\text{\AA}^2 \times 10^4$ )

H-13283-m/5

|       | x        | y        | z       | U(eq)  |
|-------|----------|----------|---------|--------|
| C(1)  | 8740(1)  | 12552(1) | 873(1)  | 269(5) |
| C(2)  | 8239(1)  | 13645(1) | 1015(1) | 269(5) |
| C(3)  | 8905(1)  | 14805(1) | 1140(2) | 322(5) |
| C(4)  | 8041(1)  | 15669(1) | 1157(2) | 345(5) |
| C(5)  | 6772(1)  | 15123(1) | 1024(2) | 325(5) |
| C(6)  | 6883(1)  | 13926(1) | 956(1)  | 282(5) |
| C(7)  | 8045(1)  | 11428(1) | 952(1)  | 257(4) |
| C(8)  | 7408(1)  | 11294(1) | 1972(1) | 271(5) |
| C(9)  | 6766(1)  | 10245(1) | 2083(2) | 284(5) |
| C(10) | 6746(1)  | 9285(1)  | 1146(2) | 286(5) |
| C(11) | 7396(1)  | 9391(1)  | 137(2)  | 318(5) |
| C(12) | 8046(1)  | 10434(1) | 53(2)   | 295(5) |
| C(13) | 5414(2)  | 8117(1)  | 2133(2) | 367(6) |
| C(14) | 10016(2) | 12438(2) | 582(2)  | 352(6) |
| O(1)  | 6147(1)  | 8211(1)  | 1164(1) | 369(4) |

\* Equivalent isotropic U defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor



Table 3. Interatomic Distances (Å) with Esd's

H-13283-m/4

10

|             |          |            |          |
|-------------|----------|------------|----------|
| C(1)-C(2)   | 1.363(2) | C(1)-C(7)  | 1.479(2) |
| C(1)-C(14)  | 1.503(2) | C(2)-C(3)  | 1.464(2) |
| C(2)-C(6)   | 1.470(2) | C(3)-C(4)  | 1.345(2) |
| C(4)-C(5)   | 1.454(2) | C(5)-C(6)  | 1.348(2) |
| C(7)-C(8)   | 1.398(2) | C(7)-C(12) | 1.407(2) |
| C(8)-C(9)   | 1.385(2) | C(9)-C(10) | 1.395(2) |
| C(10)-C(11) | 1.395(2) | C(10)-O(1) | 1.366(2) |
| C(11)-C(12) | 1.378(2) | C(13)-O(1) | 1.429(2) |

Table 4. Interatomic Angles (Deg.) with Esd's

|                   |          |                  |          |
|-------------------|----------|------------------|----------|
| C(2)-C(1)-C(7)    | 122.2(1) | C(2)-C(1)-C(14)  | 121.1(1) |
| C(7)-C(1)-C(14)   | 116.7(1) | C(1)-C(2)-C(3)   | 127.2(1) |
| C(1)-C(2)-C(6)    | 127.6(1) | C(3)-C(2)-C(6)   | 105.0(1) |
| C(2)-C(3)-C(4)    | 108.7(1) | C(3)-C(4)-C(5)   | 108.9(1) |
| C(4)-C(5)-C(6)    | 108.9(1) | C(2)-C(6)-C(5)   | 108.4(1) |
| C(1)-C(7)-C(8)    | 121.1(1) | C(1)-C(7)-C(12)  | 121.6(1) |
| C(8)-C(7)-C(12)   | 117.2(1) | C(7)-C(8)-C(9)   | 122.3(1) |
| C(8)-C(9)-C(10)   | 119.4(1) | C(9)-C(10)-C(11) | 119.3(1) |
| C(9)-C(10)-O(1)   | 124.2(1) | C(11)-C(10)-O(1) | 116.4(1) |
| C(10)-C(11)-C(12) | 120.7(1) | C(7)-C(12)-C(11) | 121.1(1) |
| C(10)-O(1)-C(13)  | 116.7(1) |                  |          |

Table 5. Anisotropic displacement coefficients ( $\text{\AA}^2 \times 10^4$ )

H-13283-m17

|       | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|-------|----------|----------|----------|----------|----------|----------|
| C(1)  | 248(7)   | 343(8)   | 201(6)   | 26(6)    | 67(5)    | 31(5)    |
| C(2)  | 280(7)   | 318(7)   | 206(6)   | -13(5)   | 89(5)    | 9(5)     |
| C(3)  | 330(8)   | 346(8)   | 281(7)   | -46(6)   | 106(6)   | -4(6)    |
| C(4)  | 430(8)   | 278(8)   | 303(7)   | -46(6)   | 110(6)   | -31(6)   |
| C(5)  | 373(8)   | 324(8)   | 280(7)   | 46(6)    | 126(6)   | -15(6)   |
| C(6)  | 289(7)   | 315(8)   | 249(7)   | 2(6)     | 111(5)   | -10(5)   |
| C(7)  | 230(6)   | 291(7)   | 237(6)   | 50(5)    | 75(5)    | 23(5)    |
| C(8)  | 306(7)   | 269(7)   | 228(7)   | 35(6)    | 91(6)    | -6(5)    |
| C(9)  | 323(7)   | 293(7)   | 259(7)   | 40(6)    | 137(6)   | 15(5)    |
| C(10) | 283(7)   | 258(7)   | 317(7)   | 33(6)    | 111(6)   | 13(5)    |
| C(11) | 349(8)   | 285(8)   | 341(8)   | 36(6)    | 155(6)   | -53(6)   |
| C(12) | 285(7)   | 348(8)   | 282(7)   | 55(6)    | 142(6)   | 6(6)     |
| C(13) | 405(8)   | 316(9)   | 430(9)   | -39(7)   | 215(7)   | -12(7)   |
| C(14) | 293(8)   | 388(9)   | 407(8)   | 46(7)    | 168(7)   | 69(7)    |
| O(1)  | 437(6)   | 263(5)   | 481(6)   | -26(4)   | 258(5)   | -54(4)   |

The anisotropic displacement exponent takes the form:

$$-2\pi^2(h^2 a^{*2} U_{11} + \dots + 2hka^*b^*U_{12})$$

Table 6. H-Atom coordinates ( $\times 10^4$ ) and isotropic displacement coefficients ( $\text{\AA}^2 \times 10^4$ )

*H-13283-m18*

|        | x         | y         | z        | U       |
|--------|-----------|-----------|----------|---------|
| H(3)   | 9851(15)  | 14895(13) | 1236(16) | 365(39) |
| H(4)   | 8227(16)  | 16536(15) | 1272(19) | 477(45) |
| H(5)   | 5953(15)  | 15554(15) | 997(17)  | 453(44) |
| H(6)   | 6174(14)  | 13344(12) | 826(15)  | 288(36) |
| H(8)   | 7438(14)  | 11943(13) | 2671(16) | 333(38) |
| H(9)   | 6373(13)  | 10197(12) | 2850(15) | 277(34) |
| H(11)  | 7361(14)  | 8713(14)  | -510(18) | 400(40) |
| H(12)  | 8514(13)  | 10488(12) | -661(16) | 288(34) |
| H(13A) | 5992(15)  | 8229(13)  | 3185(19) | 387(41) |
| H(13B) | 4625(16)  | 8714(14)  | 1814(17) | 416(41) |
| H(13C) | 5044(17)  | 7294(17)  | 1982(18) | 518(47) |
| H(14A) | 10565(17) | 11761(16) | 1092(21) | 548(49) |
| H(14B) | 10563(17) | 13187(17) | 831(20)  | 584(51) |
| H(14C) | 9802(17)  | 12288(15) | -516(21) | 533(48) |

Table 7. Hydrogen Atom Distances and Angles.

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|              |           |              |           |
|--------------|-----------|--------------|-----------|
| C(3)-H(3)    | 0.989(16) | C(4)-H(4)    | 0.987(17) |
| C(5)-H(5)    | 0.994(17) | C(6)-H(6)    | 0.973(15) |
| C(8)-H(8)    | 0.980(15) | C(9)-H(9)    | 0.977(17) |
| C(11)-H(11)  | 0.972(17) | C(12)-H(12)  | 0.991(17) |
| C(13)-H(13A) | 0.971(15) | C(13)-H(13B) | 1.031(16) |
| C(13)-H(13C) | 0.991(18) | C(14)-H(14A) | 0.970(17) |
| C(14)-H(14B) | 0.999(18) | C(14)-H(14C) | 1.002(20) |

|                     |           |                     |           |
|---------------------|-----------|---------------------|-----------|
| C(2)-C(3)-H(3)      | 123.3(9)  | C(4)-C(3)-H(3)      | 127.9(9)  |
| C(3)-C(4)-H(4)      | 126.6(10) | C(5)-C(4)-H(4)      | 124.4(10) |
| C(4)-C(5)-H(5)      | 126.0(10) | C(6)-C(5)-H(5)      | 125.1(10) |
| C(2)-C(6)-H(6)      | 125.3(9)  | C(5)-C(6)-H(6)      | 126.3(9)  |
| C(7)-C(8)-H(8)      | 120.0(10) | C(9)-C(8)-H(8)      | 117.7(10) |
| C(8)-C(9)-H(9)      | 118.2(8)  | C(10)-C(9)-H(9)     | 122.4(8)  |
| C(10)-C(11)-H(11)   | 117.9(11) | C(12)-C(11)-H(11)   | 121.4(11) |
| C(7)-C(12)-H(12)    | 119.4(8)  | C(11)-C(12)-H(12)   | 119.5(8)  |
| O(1)-C(13)-H(13A)   | 111.9(12) | O(1)-C(13)-H(13B)   | 110.7(10) |
| H(13A)-C(13)-H(13B) | 110.7(13) | O(1)-C(13)-H(13C)   | 105.2(12) |
| H(13A)-C(13)-H(13C) | 109.5(13) | H(13B)-C(13)-H(13C) | 108.7(13) |
| C(1)-C(14)-H(14A)   | 113.3(13) | C(1)-C(14)-H(14B)   | 112.3(12) |
| H(14A)-C(14)-H(14B) | 110.2(14) | C(1)-C(14)-H(14C)   | 110.2(11) |
| H(14A)-C(14)-H(14C) | 104.6(16) | H(14B)-C(14)-H(14C) | 105.7(16) |

STRUCTURE DETERMINATION SUMMARY

H-13283-m20<sup>20</sup>

Crystal Data

|                        |   |
|------------------------|---|
| Empirical Formula      | $C_{14}H_{14}O$   |
| Color; Habit           | Orange prism  |
| Crystal Size (mm)      | 0.23 x 0.30 x 0.52  |
| Crystal System         | Monoclinic  |
| Space Group            | $P2_1/c$  |
| Unit Cell Dimensions   | $a = 10.7334(9) \text{ \AA}$<br>$b = 11.1897(9) \text{ \AA}$<br>$c = 9.5808(9) \text{ \AA}$<br>$\beta = 112.265(7)^\circ$ |
| Volume                 | $1064.89(16) \text{ \AA}^3$   |
| Z                      | 4   |
| Formula weight         | 198.3   |
| Density(calc.)         | $1.237 \text{ Mg/m}^3$  |
| Absorption Coefficient | $0.071 \text{ mm}^{-1}$   |
| F(000)                 | 424   |

Data Collection

|                         |   |
|-------------------------|---|
| Diffractometer System   | Siemens R3m/V   |
| Radiation               | MoK $\alpha$ ( $\lambda$ = 0.71073 Å)                             |
| Temperature (K)         | 168   |
| Monochromator           | Highly oriented graphite crystal                                  |
| 2 $\theta$ Range        | 4.0 to 50.0°  |
| Scan Type               | $\theta$ -2 $\theta$  |
| Scan Speed              | Fixed; 3.00°/min. in $\omega$                                     |
| Scan Range ( $\omega$ ) | 1.20° plus K $\alpha$ -separation                                 |
| Background Measurement  | Estimated from 96 step profile                                    |
| Standard Reflections    | 2 measured every 98 reflections                                   |
| Index Ranges            | $-6 \leq h \leq 12$ , $-1 \leq k \leq 13$<br>$-11 \leq l \leq 11$ |
| Reflections Collected   | 2107  |
| Independent Reflections | 1791 ( $R_{\text{int}}$ = 1.7%); ( $ F_o  > 0$ )                  |
| Observed Reflections    | 1630 ( $ F_o  > 3.0\sigma( F_o )$ )                               |

Solution and Refinement

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|                                  |   |
|----------------------------------|---|
| System Used                      | Siemens SHELXTL (MicroVAX & PC Versions)  |
| Solution                         | Direct Methods  |
| Refinement Method                | Full-Matrix Least-Squares   |
| Quantity Minimized               | $\sum w( F_o  -  F_c )^2$   |
| Extinction Correction            | $\chi = 0.0117(10)$ , where<br>$F^* = F [ 1 + 0.002\chi F^2 / \sin(2\theta) ]^{-1/4}$ |
| Hydrogen Atoms                   | Refined (x,y,z) and U(iso)  |
| Weighting Scheme                 | $w^{-1} = \sigma^2( F_o ) + 0.0005( F_o )^2$  |
| Final R Indices (obs. data)      | $R_F = 3.6\%$ , $R_{wF} = 4.5\%$  |
| Goodness-of-Fit                  | 1.51  |
| Number of Variables              | 193   |
| Data-to-Parameter Ratio          | 8.4:1   |
| Largest and Mean $\Delta/\sigma$ | 0.001, < 0.001  |
| Largest Difference Peak          | 0.19 eÅ <sup>-3</sup>   |
| Largest Difference Hole          | -0.16 eÅ <sup>-3</sup>  |

References.

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1. Churchill, M. R.; Lashewycz, R. A.; Rotella, F. J. Inorg. Chem. 1977, 16, 265-271.
2. UCLA Crystallographic Computing Package, University of California Los Angeles, 1981, C. Strouse; personal communication.
3. Siemens Analytical X-Ray Instruments, Inc.; Madison, WI 1990.
4. International Tables for X-Ray Crystallography; Kynoch Press: Birmingham, England, 1974; (a) pp 99-101; (b) pp 149-150.

\* The thermal ellipsoid plot is shown at the 50% probability level.